National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2452

Hydrogen in Titanium Alloy

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and the calibration of equipment used in the determination of hydrogen in titanium alloy. SRM 2452 consists of one bottle containing 10 g of titanium alloy chips.

Certified Values: The certified value for hydrogen, expressed as mass fraction, is provided in Table 1 [1,2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [3]. The certified value is based on cold-neutron prompt-gamma activation analysis (PGAA) and volumetric measurements of hydrogen.

Table 1. Certified Value (mass fraction)

Hydrogen:	62.5 mg/kg ± 1.6 mg/kg

The uncertainty in the certified value for hydrogen is expressed as an expanded uncertainty, $U = ku_c$, calculated according to the methods in the ISO and NIST Guides [2]. The quantity u_c represents, at the level of one standard deviation, the potential combined effects of the uncertainty due to variability both within and between PGAA and volumetric measurements (combined in quadrature) and material homogeneity. The quantity $k = 2$ is the coverage factor used to obtain an expanded uncertainty with an approximate confidence level of 95 %.

Expiration of Certification: The certification of this SRM is valid until **30 April 2014**, within the measurement uncertainty specified, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). However, the certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of Certification: NIST will monitor representative portions from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was performed under the direction of R.R. Greenberg of the NIST Analytical Chemistry Division.

Preparation of SRM 2452 was performed by R.M. Lindstrom, and PGAA was performed by R.L. Paul, both of the NIST Analytical Chemistry Division.

Statistical consultation was provided by J.J. Filliben of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Steve Wise, Chief
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Certificate Issue Date: 25 April 2005

Robert L. Watters, Jr., Chief
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INSTRUCTIONS FOR USE

A minimum sample mass of 200 mg should be used for analysis. When not in use, the bottle should be kept tightly capped.

PREPARATION AND ANALYSIS

A total of 2.2 kg of titanium alloy (nominally 6 % Al and 4 % V) was taken from the same lot of chipped material intended for SRM 173c. The material was divided in half for processing. Each portion was weighed and degassed at 700 °C in a vacuum system consisting of a quartz furnace and stainless-steel high-vacuum components for 4 d to 5 d, by which time the hydrogen pressure shown by a residual gas analyzer was less than 10^{-4} Pa. After cooling to room temperature, a measured quantity of dry hydrogen was added to the system from a calibrated volume, and the material was heated to 500 °C. The reaction was completed by raising the temperature to 600 °C for 4 d to 5 d. After cooling, the residual hydrogen pressure was less than 10^{-7} Pa. The two batches were combined and passed through a No. 4 (4.75 mm) stainless-steel sieve. The material was then blended and bottled.

PGAA was used to determine the hydrogen concentration on six portions of the final material. The certified value is the sum of the residual concentration in the degassed blank material (measured by PGAA) and the quantity of hydrogen added as determined by measurements of the pressure, volume, and temperature.

REFERENCES

[1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (1995).

[2] ISO; G*uide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[3] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assessment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.